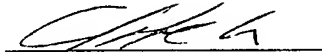


TRANSLATION ACCURACY STATEMENT

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I, Charles Edward SITCH BA,  
declare under penalty of perjury that I understand the French language and the English language; that I am conversant with the French and English languages and that, to the best of my knowledge and belief, the English language text in the attached translation of International Application No. **PCT/EP 03/50882**, including the description, claims and abstract, have the same meanings as the French language text in the original document, a copy of which I have examined.

Signature:



C. E. SITCH

For and on behalf of RWS Group Ltd

Title: Translator

Date: June 6, 2005

METHOD OF PRODUCING THICK NONLINEAR OPTICAL GRATINGS

The field of the invention is that of nonlinear optical gratings. In general, the interaction of light with an  
5 optically nonlinear material modifies its optical properties. Thus, one or more light waves are generated whose frequencies, phases or polarizations are different from those of the incident light. There are numerous applications. In particular, mention may be  
10 made of optical frequency doublers and optical frequency mixers, or optical amplifiers and optical parametric oscillators in the fields of power lasers and high-rate telecommunications.

15 The nonlinear optical effect depends on the susceptibility tensor of the material, which connects the induced polarization of the generated wave with the electric field of the incident wave. In general, this tensor of matrix form comprises 27 components called  
20 nonlinear coefficients and denoted by  $d$ .

Quadratic or 2nd-order nonlinear processes, which are the most frequently used, require phase matching between the incident wave, called the pump wave, and  
25 the wave or waves generated, called harmonic waves, during propagation in the nonlinear material. The dispersion of the optical indices between the pump wave and the harmonic waves makes it possible in practice to meet this condition only in a limited number of  
30 birefringent materials. Unfortunately, these materials do not necessarily possess the best nonlinear coefficients, wide wavelength ranges and sufficient beam focusing and operating temperature ranges.

35 The use of nonlinear optical gratings composed of structures based on nonlinear optical crystals makes it possible, under certain conditions, to partly circumvent these limitations. In particular, the technique called QPM (quasi-phase-matching) is used.

This consists in locally modifying the nonlinear properties of a nonlinear crystal so that the phase mismatch between the waves that has built up over the course of the propagation is periodically compensated (J.A. Armstrong, N. Bloembergen, J. Ducuing and P.S. Pershan, "*Interactions between light waves in a nonlinear dielectric*", Physical Review, Vol. 127, No. 6, pp. 1918-1939, 1962). In the case of ferroelectric materials, such as lithium niobate, it is known to reverse the sign of the dielectric polarization of domains a few microns in width, over the entire depth of the substrates, by applying an electric field along the Z crystallographic axis of this material. If  $d$  is the nonlinear coefficient involved, a beam propagating perpendicular to the Z crystallographic axis experiences a modulation in the susceptibility of the type  $+d/-d/+d/-d/+d/$ , etc., propitious to QPM. Depending on the desired spectral properties, it may be advantageous to use other combinations of nonlinear coefficients, with opposite or different values, with a constant or variable pitch, with a symmetrical or unsymmetrical duty cycle, with a single pitch or with successive sections of different pitches.

Certain semiconductors readily available thanks to the microelectronics industry, such as gallium arsenide (GaAs), have both high nonlinear coefficients and broad transparency ranges. However, these crystals belong to the crystallographic class of cubic symmetry, which makes them isotropic and therefore unsuitable for conventional birefringent phase matching. Moreover, they do not have ferroelectric properties that can be used for structuring a nonlinear optical grating, such as lithium niobate crystals.

However, it is possible to use GaAs in QPM mode by manufacturing structures with a periodically inverted crystal orientation. For example, it is possible to

produce monolithic stacks of GaAs plates assembled head to tail, and then to subject them to a baking operation under pressure (E. Lallier, M. Brevignon and J. Lehoux, "Efficient second-harmonic generation of a CO<sub>2</sub> laser with a quasi-phase-matched GaAs crystal", Optics Letters, Vol. 23, No. 19, pp. 1511-1513, 1998). However, it is impossible in practice to handle a large number of thin plates, and this limits the interest in such stacks.

10

Epitaxial deposition methods allow GaAs structures to be manufactured with a periodically reversed crystal orientation with fewer constraints than the above technique as regards periods and lengths of the gratings.

15

For guided wave applications, epitaxial growth of the guiding layers is possible using a seed substrate comprising an array of thin GaAs bands of reversed orientation (J.B. Yoo, R. Bhat, C. Caneau and M.A. Koza, "Quasi-phase-matched second-harmonic generation in AlGaAs waveguides with periodic domain inversion achieved by wafer-bonding", Applied Physics Letters, Vol. 66, No. 25, pp. 3410-3412, 1995).

25

For applications involving high optical power levels, it is necessary to have bulk nonlinear optical gratings several hundred microns in thickness. Epitaxial deposition techniques of the OMCVD (organometallic chemical vapor deposition) and MBE (molecular beam epitaxy) type are not appropriate. The deposition technique using a seed substrate, selective in terms of crystal orientation, called HVPE (hydride vapor phase epitaxy) may nevertheless give thick nonlinear optical gratings starting from GaAs-based structures (L. Becouarn, B. Gerard, M. Brevignon, J. Lehoux, Y. Gourdel and E. Lallier, "Second-harmonic generation of CO<sub>2</sub> laser using thick quasi-phase matched GaAs layer grown by hydride vapor phase epitaxy" Electronics

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Letters, Vol. 34, No. 25, pp. 2409-2410, 1998  
L.A. Eyres, P.J. Tourreau, T.J. Pinguet, C.B. Ebert,  
J.S. Harris, M.M. Fejér, L. Becouarn, B. Gerard and  
E. Lallier, "All-epitaxial fabrication of thick,  
5 orientation-patterned GaAs films for nonlinear optical  
frequency conversion", Applied Physics Letters,  
Vol. 79, No. 7, pp. 904-907, 2001).

10 These techniques have major drawbacks. Although the  
HVPE growth rates on the two orientations present at  
the surface of the seed substrate are very similar, a  
residual difference remains and results in a surface  
with a pronounced relief, this point being identified  
as the cause of large propagation losses.

15 Growth defects also set a limit on the quality of the  
crystals obtained: the smaller the period of the  
nonlinear optical gratings, the more difficult it  
becomes to obtain these crystals faithfully with the  
20 seed substrate over a large thickness.

The method proposed by the invention makes it possible  
either to obtain a nonlinear optical grating of high  
quality over substantial thicknesses or to produce  
25 waveguides that include a nonlinear optical grating  
without substantial attenuation. This method applies  
most particularly to gratings based on semiconductor  
materials such as GaAs, which have major technical  
advantages both from the standpoint of their physical  
30 properties and their technology employed.

More precisely, the subject of the invention is a  
method of producing a thick nonlinear optical grating  
from an initial thick nonlinear optical grating, the  
35 thickness of the nonlinear optical grating being  
greater than the thickness of the initial nonlinear  
optical grating, said initial grating comprising at  
least one plurality of mutually parallel plane layers,  
said layers having at least two nonlinear coefficients

having algebraically different values, said initial grating having a first face and a second face that are approximately parallel to each other and approximately perpendicular to the mean plane of the layers, and said  
5 second face being free, characterized in that it comprises the following production steps:

- a first step of determining the thickness of that upper part of the initial grating which lies beneath the second face, which upper part has  
10 structural imperfections;

- a second step of polishing the second face of said initial grating, making it possible to remove the upper part having said imperfections and to obtain a polished and plane third face, said face approximately  
15 perpendicular to the mean plane of the layers;

- a third step of cleaning and checking said third face; and

- at least a fourth step of epitaxially depositing at least one layer of material deposited on  
20 said third face, the epitaxial growth reproducing, in said deposited layer, a structure similar to that of the initial grating, the combination of the initial grating and said deposited layer constituting the nonlinear optical grating.

25

The invention will be more clearly understood and other advantages will become apparent on reading the following description given by way of nonlimiting example and thanks to the appended figures in which:

30 - figures 1 to 4 show the various steps of the production method according to the invention. They comprise a general sectional view of the grating and an encircled enlarged partial view showing the structure of the grating;

35 - figures 5a and 5b show the various steps of the production of the first optical grating, in the case in which it is made from a monolithic assembly of crystalline plates; and

- figures 6a to 6c show the various steps of a

method of preparing the grating that makes it easier to carry out the operation of polishing the initial optical grating.

5 Figure 1 is a view in section and an enlarged view of the initial nonlinear optical grating 1. This grating 1 has a plurality of layers 20 whose linear coefficients vary from one layer to the next layer. This grating has a first face 11 and a second face 12 that are  
10 approximately parallel. There are various methods of producing said initial grating.

In a first embodiment, as shown in figure 1, the nonlinear material is deposited on a seed substrate 3.  
15 This comprises a crystalline material having a first crystal orientation, the upper face 11 of the seed substrate having a thin structure, said structure being formed from what is called the precursor grating of parallel bands of the same crystalline material but of  
20 reverse crystal orientation to that of the crystalline material of the seed substrate 3. The deposition is carried out, for example, by the HVPE epitaxial growth method on the upper face 11 of the seed substrate. In this case, the deposition, which is selective in terms  
25 of crystal orientation, is carried out over a total thickness  $E_0$ . This thickness includes, on the one hand, imperfections on the surface 12 over a first thickness  $E_{01}$  and, on the other hand, structural imperfections over a second thickness  $E_{02}$  that are due, for example,  
30 to the variations in growth rate of the various layers during deposition or due to initial defects in the seed substrate. The useful thickness  $E_1$  is therefore given by:

$$E_1 = E_0 - E_{01} - E_{02}.$$

35

In a second embodiment, the method of producing the initial optical grating comprises the following steps:

- a first substep of producing a stack of crystalline plates 21 having plane parallel faces, of

the same material, of small thickness and of periodically alternating crystal orientation; and

• a second substep of assembling said plates so as to obtain a single monolithic assembly constituting said initial optical grating 1, said grating having a first face 11 and a second face 12 that are approximately perpendicular to the mean plane of the crystalline plates as shown in figure 5a.

Here again, it is impossible to obtain a perfect stack over its entire thickness, and consequently the faces of the stack also have surface and structural imperfections.

The first step of the production method consists in determining the useful thickness  $E_1$ . Since the thicknesses of the various layers are of the order of a few microns to a few tens of microns, the means of determining the thickness, including the imperfections, are optical display devices. Observation via the edge of the initial grating allows the thicknesses  $E_0$ ,  $E_{01}$  and  $E_{02}$  to be determined. Optionally, a cut may be made into the substrate so that the grating is flush with the substrate after cutting, which thus improves the observation. A chemical development operation may also be carried out, so as to improve the contrast.

Before the second step of the production method is carried out, preliminary preparation steps may be performed so as to make said step easier.

These preliminary steps are shown in figures 6a to 6c in the case in which the initial grating is produced on a seed substrate. They comprise:

• a first preliminary step of polishing the lower face 14 of the substrate in order to obtain the plane polished face 14a (figure 6a and figure 6b); and

• a second preliminary step of bonding at least said lower face 14a to at least one plane support



(figure 6c), the fitting of the support making it easier to handle the initial optical grating for the subsequent polishing operations.

- 5 The preliminary steps in the case in which the initial grating is a monolithic stack of crystalline plates are the following:
- a first preliminary step of polishing the first face 11 of the monolithic stack in order to obtain the  
10 plane face 11a; and
  - a second preliminary step of bonding at least said first face 11a to at least one plane support 32 as indicated in figure 5b, the fitting of the support making it easier to handle the monolithic assembly for  
15 the subsequent operations of polishing the second face. Optionally, lateral reinforcements 31 may be placed on at least one side of the monolithic stack 2, said reinforcements being bonded to the support 32.
- 20 The second step of the production method consists, firstly, in polishing the face 12 by mechanical abrasion so as to remove the surface perturbations present in the thicknesses  $E_{01}$  and  $E_{02}$  and secondly in then supplementing this first polishing operation with  
25 a second, chemical-mechanical polishing operation and in obtaining sufficient surface quality to carry out epitaxial deposition. The polished plane face 13 approximately perpendicular to the mean plane of the layers of the grating 1 is therefore obtained. This  
30 second operation may prove to be superfluous under certain epitaxial deposition conditions using the HVPE method.

When the first grating has been produced on a large  
35 substrate, typically with a diameter greater than 50 millimeters (i.e. equivalent to a standard wafer diameter of 2 inches), it is possible to polish the face 12 of the grating and the lower face 14 of the substrate simultaneously in a twin-platen polisher.

These polishers are used in particular in the microelectronics industry to polish semiconductor wafers. However, in this case it is more difficult to control the thickness and the quality of the grating obtained. Therefore the procedure is to carry out successive steps until a polished grating no longer containing structural perturbations is obtained.

In order for this polishing operation to be performed successfully, the grating must not be too fragile. When the initial grating has been produced on a substrate, a minimum thickness of 50 microns for the grating alone and 300 microns for the substrate for is desirable; when the initial grating does not include a substrate, a minimum thickness of 350 microns is desirable.

What is obtained at the end is the grating shown in figure 2. The second face 12 has become the third, plane face 13. The thickness of the grating is now  $E_1$ .

In a third step of the method, the surface 13 is cleaned and checked, for example by optical means, so as to confirm that the grating is ready to be used for epitaxial deposition under conditions that will preserve the structure of the grating.

Finally, in a fourth step of the method, at least a first layer 1a of material is deposited on said third face 13 under conditions that preserve the structure of the first grating, the combination of the first grating 1 and the said layer 1a constituting the second nonlinear optical grating 2, as indicated in figure 3. Since the layer 1a has a thickness  $E_{1a}$ , the final thickness of the grating will now be  $E_2$ , where:

$$E_2 = E_{1a} + E_1.$$

Of course, it is possible to repeat the fourth step at least once, as indicated in figure 4. In this figure, a second layer 1b of thickness  $E_{1b}$  is deposited on the

layer 1a. In the end, the grating therefore has a total thickness  $E_{2a}$  given by:

$$E_{2a} = E_2 + E_{1b} = E_{1b} + E_{1a} + E_1.$$

5 This technique may be used:

- either to increase the final thickness of the grating, each layer then being composed of the same material;
  - or to produce particular functions of the
- 10 waveguide type, the materials of two successive layers then having different optical properties such as, in particular, their optical index.

The layers are deposited by epitaxy. Among possible  
15 methods, mention may be made of:

- the OMCVD epitaxial growth method and the MBE epitaxial growth method for producing layers not exceeding a few microns in thickness; and
  - the HVPE epitaxial growth method for producing
- 20 thicker layers.

If the starting substrate includes zones with no nonlinear optical grating, the quality with which its surface is prepared may also make it possible to  
25 fabricate by epitaxy, using the OMCVD or MBE methods, structures that are co-integrated with nonlinear optical grating waveguides, such as for example laser diodes, optical modulators, Bragg grating sections, etc.

30

There are various possible materials allowing nonlinear optical gratings to be produced. For example, it is possible to use a crystal belonging to the  $\bar{4}3m$  crystallographic class.

35

The susceptibility tensor for this crystallographic class is given below:

$$\begin{pmatrix} 0 & 0 & 0 & d & 0 & 0 \\ 0 & 0 & 0 & 0 & d & 0 \\ 0 & 0 & 0 & 0 & 0 & d \end{pmatrix}$$

Such a crystal may be a gallium arsenide (GaAs) or  
indium phosphide (InP) or cadmium telluride (CdTe) or  
zinc selenide (ZnSe) or zinc telluride (ZnTe) or  
5 gallium phosphide (GaP) or indium arsenide (InAs) or  
indium antimonide (InSb) crystal.

The material may also be tellurium (Te) or selenium  
(Se) or gallium nitride (GaN).

10

It is also possible to use gallium aluminum arsenide  
(GaAlAs) for producing layers of different indices for  
producing waveguides.